

# Sieve Test of Metal Powders

By Rolla E. Pollard

Reproducible results were obtained in sieve tests of sponge iron, electrolytic iron, electrolytic copper, and nickel when certain variables affecting the sieving characteristics of the powders were eliminated or controlled. One of these was a cumulative sampling error resulting from repeated riffle cutting of limited powder supplies. Another was the effect of exposure of the powder to humid atmospheres. The effects of both variables were demonstrated in tests with sponge iron. Variations of considerable magnitude were observed when either variable was uncontrolled.

Significant variations also were noted when the same powders were sieved with different sets of certified sieves. These differences were reduced considerably in magnitude when comparisons were made on the basis of the size of the average opening as determined during the certification tests instead of the nominal sieve opening.

Development of more effective methods for controlling these variables or evaluating their effects would improve the reproducibility of sieve tests.

## I. Introduction

In most arts and industries producing or using powdered material, close control of the size distribution of particles is necessary. Sieving is the most rapid and convenient method of size analysis, but it is limited to particles that are retained on a 325 mesh screen, having a nominal opening of 44  $\mu$ . Manufacturing difficulties have prevented production of sieves of smaller mesh, hence the size distribution of finer materials must be determined by other means.

Two standard sieve series, with specified dimensions and permissible variations, have been widely used in this country for many years. The Tyler Standard Scale introduced in 1910 is based on the size of the average opening, 0.0029 in., in 200 mesh wire cloth. The openings of the other sieves in the series vary by the fixed ratio of the square root of two. The U. S. Standard sieve series, proposed by the National Bureau of Standards in 1919, is based on a 1 mm (0.0394 in.) opening in the No. 18 sieve. In general, the sieve sizes vary in the same ratios as the Tyler Series and there is little difference between corresponding sieves. The U. S. Standard Sieve Series has been adopted by the U. S. Government

[1],<sup>1</sup> by the National Bureau of Standards [2], by the American Society for Testing Materials [3], and by the American Standards Association [4]. A calibration service is available at the National Bureau of Standards where, for a small fee, sieves may be tested and, if acceptable, certified to conform to the requirements of these specifications.

Standard methods have been adopted by the American Society for Testing Materials and other technical organizations for sieve analyses of particular materials such as ores [4], refractories [5], fine and coarse aggregates [6], pigments [7], powdered coal [8], soap [9], cement [10], and roofing materials [11, 12]. Three of these specifications, namely those for powdered coal [8] and roofing materials [11] and [12] include criteria for the reproducibility of sieving tests by these standard methods. All three require that duplicate determinations by the same operator, using the same sieves, shall check within 1 percent of the total weight of the sample on all sieves. The standard method for coal [8] requires that duplicate determinations by different operators using different sieves shall check within 3 percent on the material sieved on the No. 200 sieve.

<sup>1</sup> Figures in brackets indicate the literature references at the end of this paper.

Tentative standard procedures for sieve analysis of granular metal powders have recently been proposed by the American Society for Testing Materials (B214-46T) and the Metal Powder Association (5-46T). Considerable information on sieving is available in the literature, but most of it is concerned with materials other than metal powders. Investigators generally agree that the sieving characteristics of different materials vary considerably. In particular, the rate of sieving, sharpness of separation, and average particle size of sieve fractions are influenced by the shape of the particles and their size distribution. The diversity of methods used in the manufacture of metal powders causes wide variations in particle shape and particle size distribution. Consequently different metal powders or powders of the same composition made by different processes, might be expected to display variations in sieving characteristics.

It is generally agreed that satisfactorily reproducible sieving results can be obtained for a given material when a single set of sieves and a standard method of testing are used. If the sieving conditions are not closely controlled, or if different sets of sieves are used, variations considerably greater than those cited as criteria for reproducibility may be obtained. For example, in cooperative tests of standard samples of cement, Wig and Pearson [13] found differences as high as 13 percent in the values reported from 80 different laboratories, all using standard sieves. Similar differences have been reported for cooperative tests of foundry sand [14], powdered coal [15] and road building materials [16]. Weber and Moran [21] in tests with granular and powdered sodium bicarbonate obtained differences of as much as 12

percent in sieving, values obtained with different standard sieves in the same laboratory.

In beginning an investigation of the basic principles of powder metallurgy at the National Bureau of Standards, one of the first objectives was to determine the effects of particle size distribution on the properties of metal powders and the compacts made from them. In the process of accumulating supplies of sieved fractions of various powders for these studies, significant variations in sieve analyses were noted when samples of the same powder were sieved at different times with the same sieves. Variations of considerable magnitude also were obtained when different sets of certified sieves were used for the same powder.

Therefore, further study of the problems connected with the sieve analysis of these powders seemed desirable. The results presented herein are those obtained in preliminary studies of the effect of variation in sieving time, the reproducibility of sieve analyses and the particle size distribution within the sieve fractions.

## II. Materials

The character and properties of metal powders used in obtaining the data presented herein are given in table 1. Four different methods of production are represented; reduction (iron), electrolysis (iron and copper), pulverization (nickel), and atomization (zinc, tin, and lead).

The size distribution of the sponge iron, electrolytic iron, copper and nickel powders was found to be such that several fractions of substantial size could be obtained by sieving. These powders,

TABLE 1.—*Properties of the metal powders*

Material	Method of production	Fineness (approximate)	Apparent density	Flow rate <sup>a</sup>
			gm/cc	sec
Sponge iron.....	Reduced mill scale pulverized, screened and annealed.	32 to 40 percent through 325 mesh.....	2.51	36
Electrolytic iron.....	Electrodeposit in sheet form pulverized, screened, annealed, and again screened.	37 to 46 percent through 325 mesh.....	2.28	47
Electrolytic copper.....	Electrolytic "Grade B".....	51 to 59 percent through 325 mesh.....	2.70	31
Nickel.....	Mechanical pulverization.....	68 to 73 percent through 325 mesh.....	3.65	29
Zinc.....	Atomization.....	99 percent through 325 mesh.....	2.02	No flow
Tin.....	do.....	89 percent through 325 mesh.....	4.19	No flow
Lead.....	do.....	84 percent through 325 mesh.....	5.61	No flow

<sup>a</sup> Hall Standard Flowmeter—50-gram sample.

therefore, were suitable for studies of the effect of sieving time and the reproducibility of sieve analyses. The atomized powders on hand at the time were not suitable for such studies because the powders were extremely fine and sieving produced only one or two small fractions. However, microscopic measurements of accumulated fractions gave useful information on particle size distribution within fractions.

Microscopic examination revealed that the sponge iron, which had been prepared by reduction of millscale, was composed of irregular plate-like particles as shown in figure 1. The thickness of these particles appeared to be considerably less than either of the other two dimensions as indicated by the relatively small adjustment required to change the focus from the top to the bottom of the particle. Many of the larger particles consisted of several plates apparently held together by oxide (fig. 2). The pearlite areas frequently observed indicated the presence of carbon in the metal.

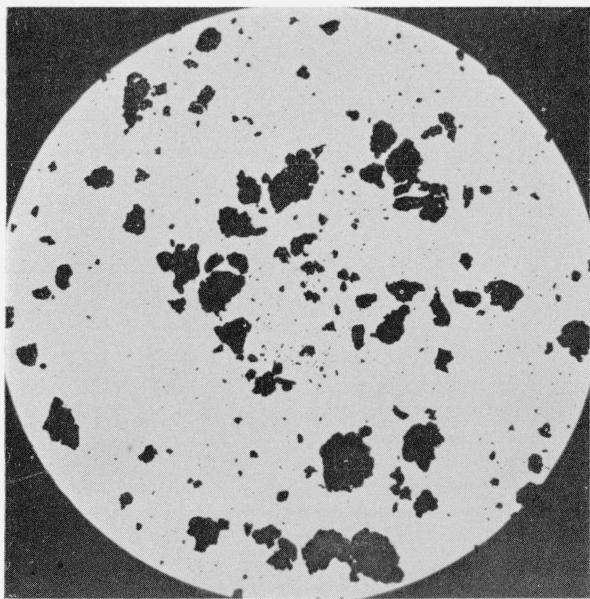


FIGURE 1.—*Sponge iron powder particles, as received.*  $\times 50$ .

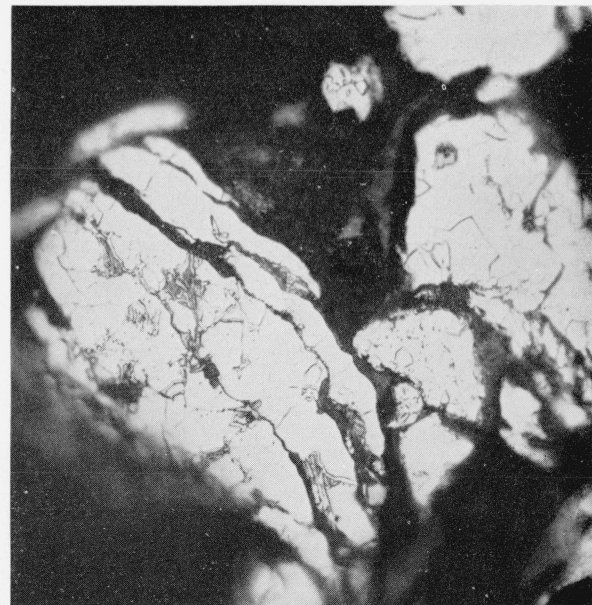


FIGURE 2.—*Areas of pearlite are visible in sections of sponge iron particles.*

Some particles appear to be composed of several plates cemented together with oxide. Etched with nital.  $\times 500$ .

The electrolytic iron was similar to the sponge iron in shape of particle and oxide content, but the plates were more distorted and there was no microscopic evidence of carbon in the iron (figs. 3 and 4).

The electrolytic copper powder was composed of two distinctly different types of particles. One was plate-like, and the other was irregular and tree-like in shape, as shown in figure 5, 6, and 7. Many of the spaces between the branches in the tree-like particles appeared to be filled with light blue or dark blue oxide.

The nickel particles (figs. 8, 9, and 10) were roughly rectangular or elliptical in shape. Plate-like particles were prevalent, and the larger particles often consisted of two or more plates apparently cemented by oxide.

The zinc, tin, and lead powders, produced by atomization were composed of globular particles, all dimensions being of the same order of magnitude. These particles appeared to be largely solid metal with little porosity and few inclusions (figs. 11 to 16, inclusive).

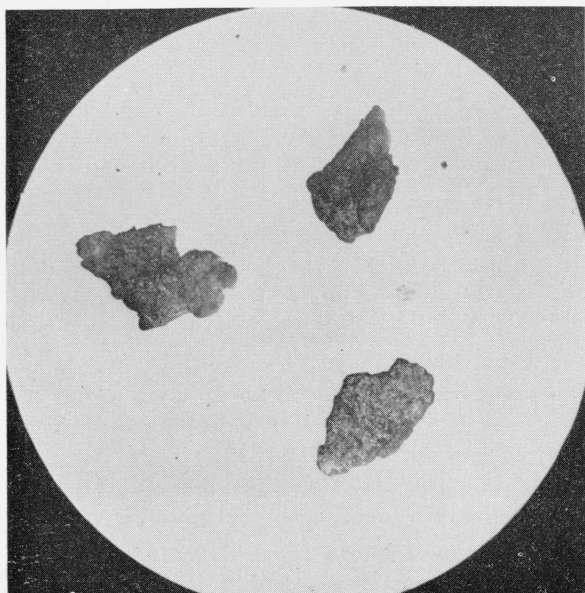


FIGURE 3.—*Electrolytic iron particles retained on 140 mesh sieve.  $\times 50$ .*

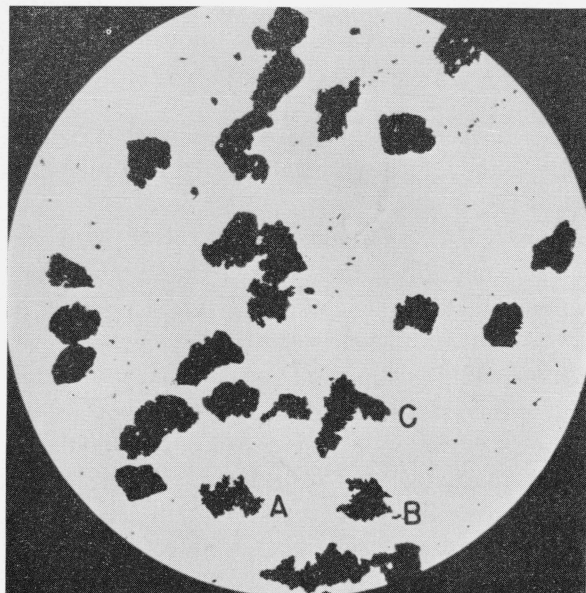


FIGURE 5.—*Electrolytic copper particles retained on 200 mesh sieve.  $\times 50$ .*

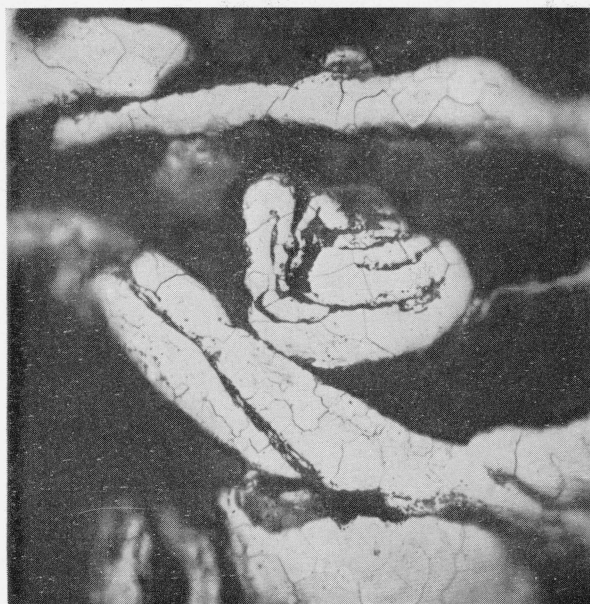


FIGURE 4.—*Sections of electrolytic iron particles which show distortion as a result of pulverization. Some particles consist of plates held together by oxide. Etched with nital.  $\times 500$*

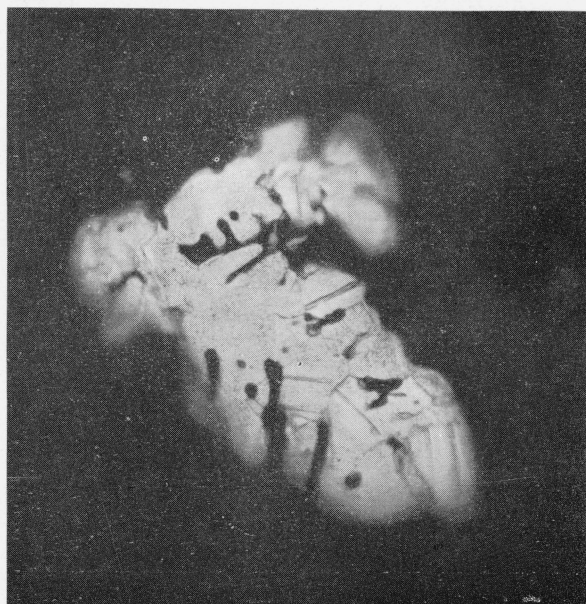


FIGURE 6.—*Sections of irregular, tree-like copper particles. Some of the spaces between branches apparently are filled with oxide. Etched with ammonium hydroxide and hydrogen peroxide.  $\times 500$ .*





FIGURE 7.—Spaces between branches of tree-like, electrolytic copper are filled with oxide.

Same etchant as figure 6.  $\times 500$ .

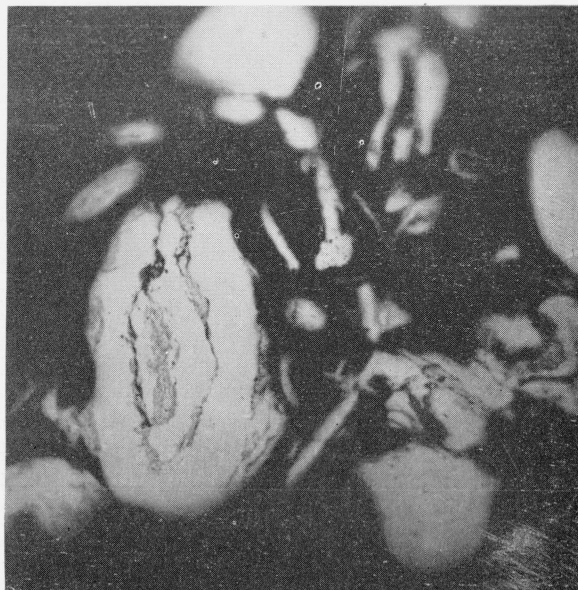


FIGURE 9.—Sections of plate-like nickel particles.

Some of the particles were distorted during pulverization and the interspaces apparently filled with oxide. Etched with nitric and acetic acids in acetone.  $\times 500$ .

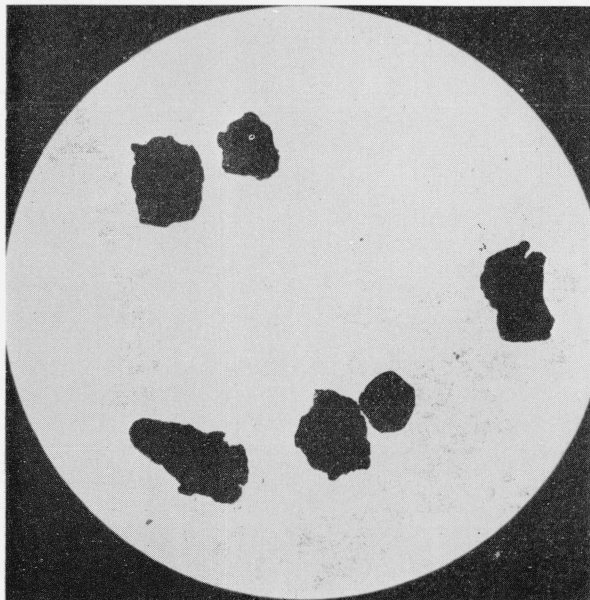


FIGURE 8.—Nickel powder particles retained on 140 mesh sieve.  $\times 50$ .



FIGURE 10.—Section of nickel particle that apparently consists of several plates cemented together with oxide.

Same etchant as figure 9.  $\times 500$ .

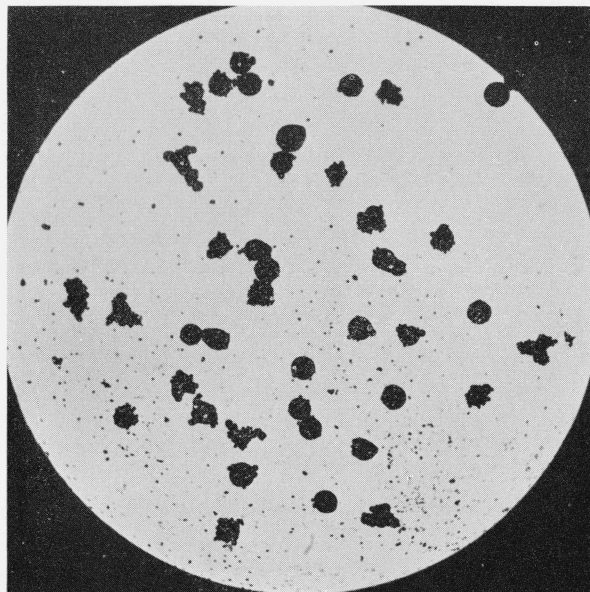


FIGURE 11.—*Particles of atomized zinc powder retained on 325 mesh sieve.  $\times 50$ .*

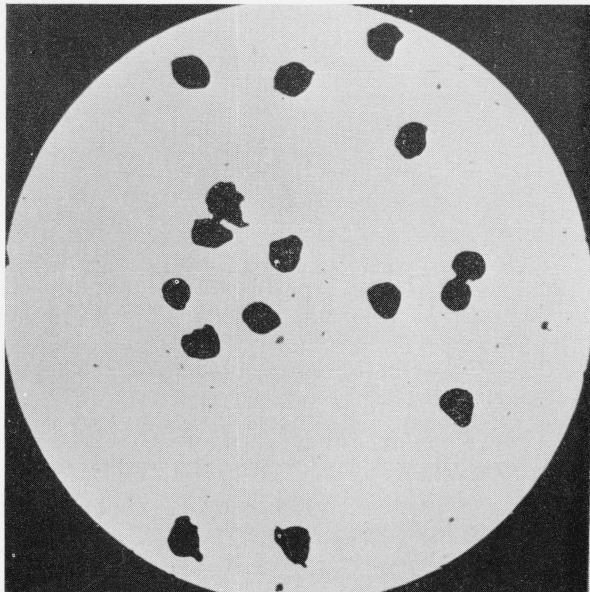


FIGURE 13. *Particles of atomized tin powder retained on 200 mesh sieve.  $\times 50$ .*

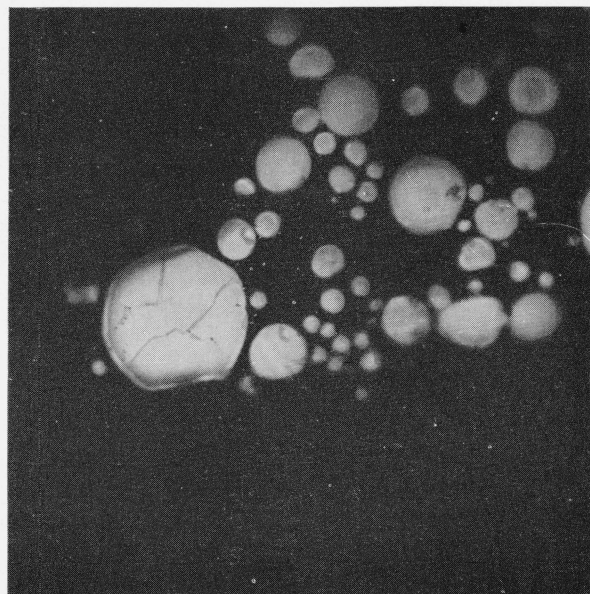


FIGURE 12.—*Sections of globular zinc particles show few voids or inclusions.*  
Etched with nitric acid in amyl alcohol.  $\times 500$ .

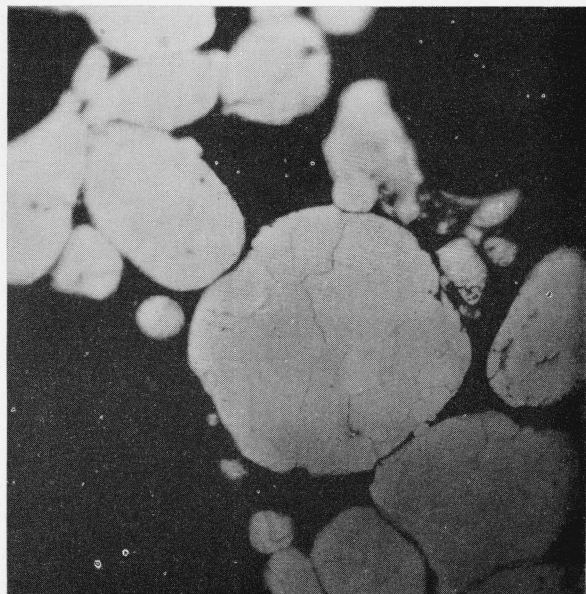


FIGURE 14. *The globular particles of tin contain few voids or inclusions.*  
The sections were etched with nitric and acetic acids in glycerol.  $\times 500$ .

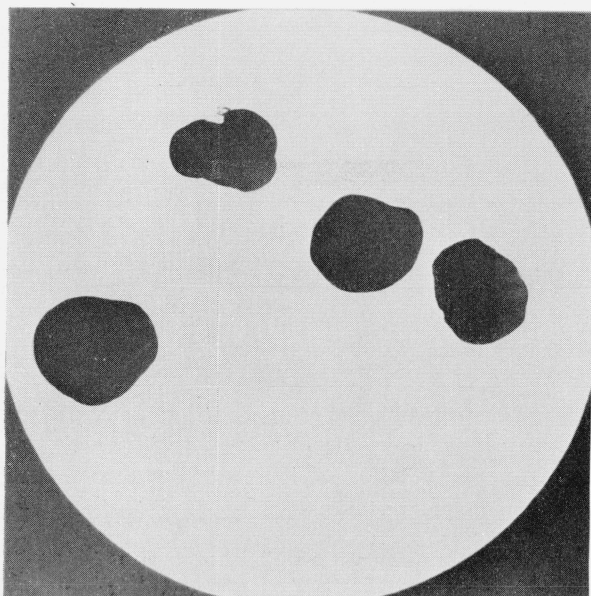


FIGURE 15. *Particles of atomized lead powder retained on 80 mesh sieve.  $\times 50$ .*

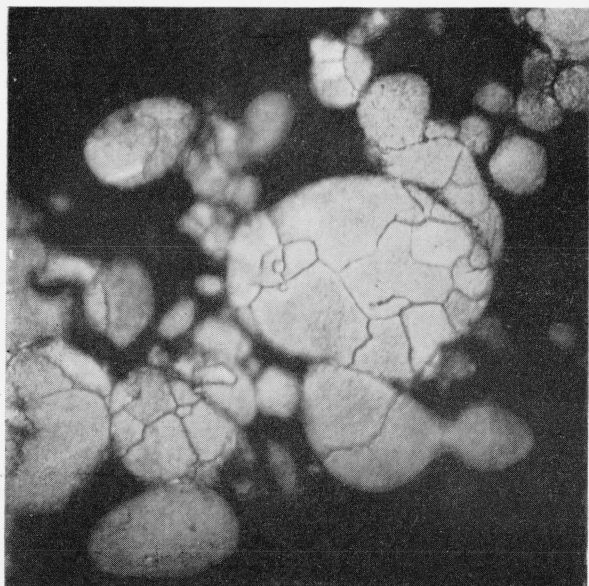


FIGURE 16. *Lead particles are roughly spherical with few voids or inclusions.*

Sections etched with nitric and acetic acids in glycerol.  $\times 500$ .

### III. Method of Testing

A Tyler RO-Tap shaker provided with an automatic time switch was used in making the sieve tests as specified in ASTM B-46T and MPA4-45T Standards. The machine was adjusted to accommodate a set of five tested sieves,

which were selected from the following six sieves of the U. S. Standard Series:

Sieve No.	Average sieve opening	Permissible variation in average opening	Maximum permissible opening microns	Permissible dimension, which not more than 5 percent of the opening are permitted to exceed
	$\mu$	$\mu \pm$		
80	177	11	247	212
100	149	9	208	179
140	105	6	147	126
200	74	5	118	96
230	62	4	117	90
325	44	3	83.6	64

Two sets of sieves, designated as set I and set II were used for most of the tests. The No. 230 sieve was omitted from both of these sets. In later tests set No. III was used, which omitted the No. 80 but included a No. 230 sieve. All sieves were tested at the National Bureau of Standards and certified to conform to ASTM Specification E11-9 and Federal Specification RR-S366a.

The sieves were brushed with a stiff bristle brush after each test. At frequent intervals, for instance at the beginning of a series of tests or when a different material was to be sieved, the sieves were cleaned in hot soapy water with stiff bristle brushes, rinsed with alcohol and dried in an oven at  $110^{\circ}\text{C}$ .

The sampling procedure included the use of a riffle-type sample splitter, to reduce the entire supply of each metal powder (50 to 100 lb in most cases) to a "sample supply" that could be stored in one to five 1-pint Mason jars (3 to 15 lb). Smaller samples for individual tests were taken from these jars, after thorough shaking, by means of a curved spatula ("scoopula"), which reached to the bottom of the container.

### IV. Time of Sieving

To determine the effect of shaking time on sieving, 25, 50, 100, and 150 gram samples of sponge iron powder (3 samples of each weight) were sieved for various periods of time, using Sieve set I. Two series of tests were made, one in March, the other in July 1946. The schedules of sieving time periods for both series are given in table 2.

To obtain information on possible differences caused by sampling, each of the three samples of



TABLE 2.—Schedules of sieving time for two series of sieve tests of sponge iron—Sieve Set I

Weight of sample	Number of samples	Time of sieving and resieving
SERIES I		
<i>g</i>		<i>minutes</i>
25	3	10, 20, 30, 40, 10.
50	3	5, 10, 20, 30, 40, 60, 20.
100	3	5, 10, 20, 30, 40, 60, 80, 30.
150	3	5, 10, 20, 30, 40, 60, 80, 100, 40.
SERIES II		
25	3	5, 10, 20, 30, 40, 60, 80, 100, 120, 10.
50	3	5, 10, 20, 30, 40, 60, 80, 100, 120, 20.
100	3	5, 10, 20, 30, 40, 60, 80, 100, 120, 30.
150	3	5, 10, 20, 30, 40, 60, 80, 100, 120, 40.

each weight was resieved through the selected range of time periods. After each period the weighed sieve fractions were thoroughly mixed and resieved for the next time period. The total weight added to the top sieve at the beginning of each period was used in calculating the percentage by weight retained by the sieves during that period.

The last time period in each series of the schedule was selected as a duplication of one of the earlier periods for the purpose of determining to what extent disintegration of particles had occurred as a result of grinding or abrasion during prolonged sieving. The total time of sieving, including the last or duplicate periods ranged from 110 to 505 minutes.

The results of two series of sieve tests on sponge iron are shown in figures 17, 18, 19, and 20. The sieving rate was greatest during the initial period, decreased rapidly for periods up to 15 to 30 minutes, and then decreased slowly with continued sieving time. Appreciable amounts of material continued to pass through the sieves after sieving periods up to 120 minutes. It is evident, therefore, that for practical purposes at least, there is no definite time of sieving at which complete separation can be said to have occurred.

The period of high sieving rate or rapid change in weight distribution represents the time required for the definitely undersized particles to pass through the sieve openings. The period of slowly diminishing sieving rate represents the continued separation of the so-called "difficult" particles

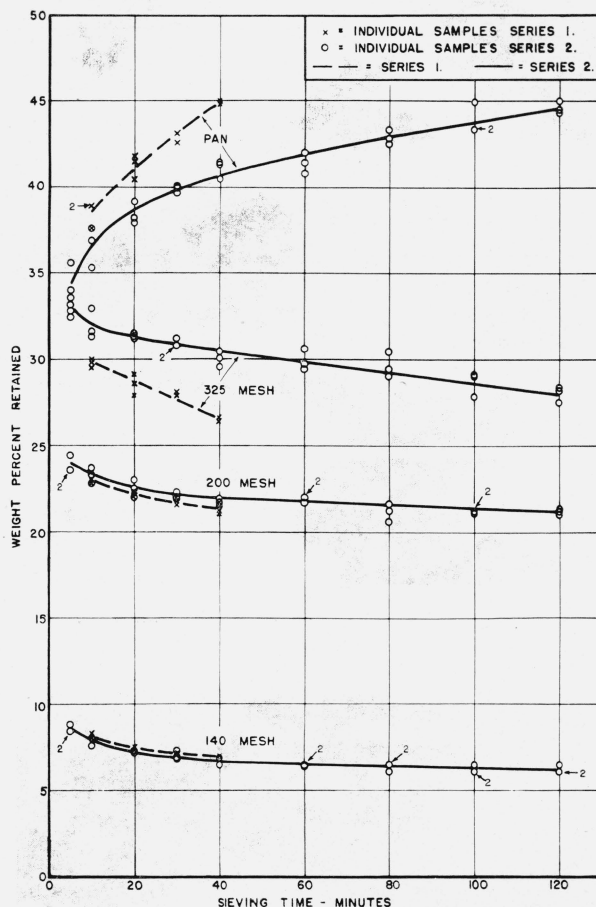


FIGURE 17.—Sieve tests of 25-gram samples of sponge iron made at different times using sieving schedule given in table 2.

Series I was made in March, Series II in July.

(particles whose controlling dimensions are close to those of the sieve openings or which, because of their shape, pass through the openings with difficulty) or the continued passage of larger particles through oversize openings.

The values for the duplicate time periods, given in table 3, indicate that the continuous change in weight distribution during prolonged sieving was not caused by disintegration of the particles. The values agree within the range of differences between samples, although the sieving time between duplicate periods was as much as 450 minutes (see sieving schedule, table 2). Evidently disintegration of these sponge-iron particles as a result of grinding or abrasion, during prolonged sieving, is negligible.

The average total sieve losses during the sieving



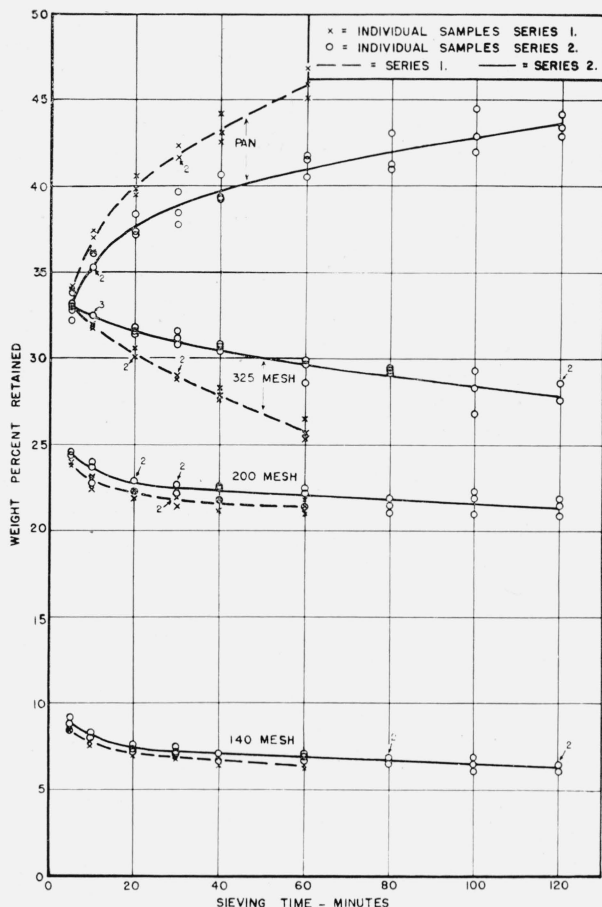


FIGURE 18.—Sieve tests of 50-gram samples of sponge iron.

schedules, for the 25, 50, 100, and 150 gram samples, respectively, were 1.0, 1.5, 1.2, and 1.0 percent for Series I and 2.0, 1.9, 1.3, and 0.8 for Series II. Each of these values represents the accumulative losses over as many as 10 different sieving periods, hence these losses are not considered to be excessive. As the total losses decreased appreciably with increase in weight of sample, particularly in Series II where the sieving schedules were approximately equal, they probably resulted from repeated handling rather than from formation of appreciable amounts of dust.

### V. Reproducibility

The curves of figures 17, 18, 19, and 20, which represent the change in sieving rate with increasing sieving time are noticeably different for Series I and Series II. This difference is particularly marked for the pan fractions that are measures of the material passing all of the sieves. The differ-

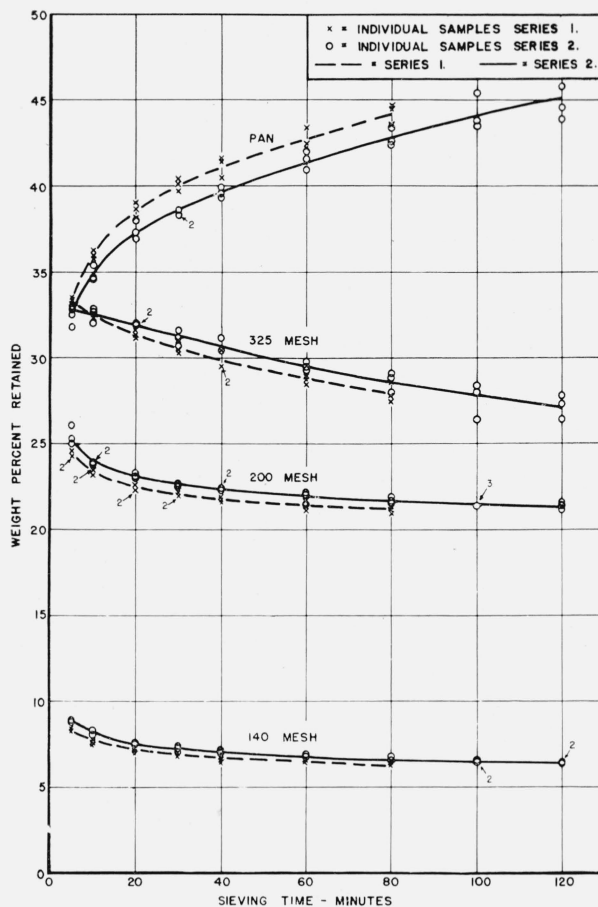


FIGURE 19.—Sieve tests of 100-gram samples of sponge iron.

ence between the pan fractions of the two series, therefore, represents the algebraic sum of the differences between the corresponding sieve fractions. The values for the corresponding sieve samples of the two series fall into definitely separate groups after the initial 5-minute sieving period. With increased sieving time the mean values for the two series are separated by an amount greater than the difference between individual samples of either series.

The results obtained for Series I show differences between individual samples of the same weight, sieved for the same length of time, ranging from 0 to a maximum of 1.9 percent of the total weight of the sample. In Series II the differences between corresponding samples range from 0 to a maximum of 2.4 percent. The differences between the average values for the two series vary from 0 to a maximum of 4.6 percent. It should be noted that these differences are con-

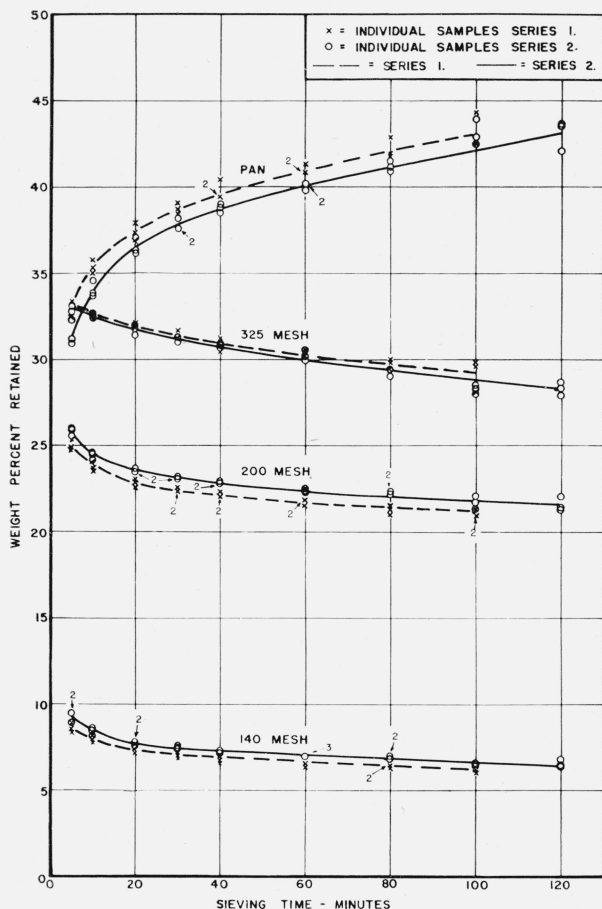


FIGURE 20.—Sieve tests of 150-gram samples of sponge iron.

sistently in the same direction. In other words, there is no overlapping of results between the two series, and the maximum scatter of corresponding values for both series considered together (5.3 percent) is more than twice as great as that of either series alone (1.9 to 2.4 percent).

The close grouping of the values for the samples of each series and the fact that smooth curves can be drawn through the points representing them indicate that testing conditions were relatively stable during each series of tests. The separation of the curves by an amount greater than the scatter between the samples indicates that testing conditions or the sieving characteristics of the powder were different for the two series. The differences between the corresponding fractions of the same series are comparatively small and can be attributed chiefly to the sampling error inherent in the method used in selecting the samples from the "sample supply". The

differences between the two series are much greater in magnitude and are consistently in the same direction. It must be concluded, therefore, that they were caused by other uncontrolled variables.

One of these variables would be an additional sampling error resulting from the use of a sample splitter to cut the powder supply, originally 100 lb, to the two different "sample supplies" of 3 to 15 lb from which the samples for the two series were taken. This error, being produced by a mechanical operation, would be expected to be smaller than that resulting from the manual method used in selecting samples from the "sample supply".

Another possible variable is suggested by the different seasons of the year in which the tests were made, one in March, the other in July 1946. The physical condition or sieving characteristics of the powder may have been changed by exposure to different atmospheric conditions. A change in size distribution, for instance, could be produced under oxidizing or corrosive conditions such as would prevail with high humidities, by the formation of additional composite particles similar to those previously described as being composed of two or more smaller particles cemented together by oxide (see figs. 2, 4, 6, 9, and 10). Variations in humidity might also affect the sieving characteristics of the powder by changing the tendency of particles to agglomerate, or by modifying the static charges accumulated during handling or sieving.

To test the effect of these and other possible variables such as clogging of sieves or changes in sieve motion, additional sieve tests were made in November and December 1946, and January and February 1947. For these tests sieve set No. III was used.

A new hard maple plug was placed in the RO-Tap machine, and the sieve motion was checked at frequent intervals over the 4-month period by counting the number of hammer blows per minute. The rate of hammer blows was found to average 154 per minute with variations no greater than  $\pm 1$  blow per minute. This fixed the speed of rotary motion of the sieves at  $289 \pm 2$  revolutions per minute.

Samples of sponge iron 25, 50, and 100 grams in weight, all taken from the same freshly riffle cut "sample supply", were sieved for periods of

TABLE 3.—*Effect of prolonged sieving of sponge iron*

[Samples were sieved and resieved to schedules given in table 2, Sieve Set I]

Sieve No.	Weight percent retained															
	25-gram samples				50-gram samples				100-gram samples				150-gram samples			
	First 10-min period		Last 10-min period		First 20-min period		Last 20-min period		First 30-min period		Last 30-min period		First 40-min period		Last 40-min period	
	Mean 3 samples	Deviation from mean	Mean 3 samples	Deviation from mean	Mean 3 samples	Deviation from mean	Mean 3 samples	Deviation from mean	Mean 3 samples	Deviation from mean	Mean 3 samples	Deviation from mean	Mean 3 samples	Deviation from mean	Mean 3 samples	Deviation from mean
SERIES I																
80.....	Trace	-----	Trace	-----	Trace	-----	Trace	-----	Trace	-----	Trace	-----	Trace	-----	Trace	-----
100.....	Trace	-----	Trace	-----	Trace	-----	Trace	-----	Trace	-----	Trace	-----	Trace	-----	Trace	-----
140.....	8.1	±0.2	7.9	±0.1-0.2	7.1	±0.1	7.2	±0.1-0.3	6.9	±0.1	6.8	±0.1-0	6.9	±0.1-0.2	6.8	±0.2
200.....	23.0	+ .4-0.2	22.8	+ .1- .2	22.2	+ .1-0.3	22.3	+ .2- .3	22.1	± .1	22.0	± .4	22.2	± .1	21.8	± .2
325.....	29.8	+ .2- .3	30.0	+ .2- .3	30.2	+ .4- .1	30.3	± .1	30.6	+ .3-0.2	30.6	+ .3-0.4	30.9	+ .3- .5	30.8	+ .4-0.7
Pan.....	38.5	+ .4- .9	39.1	+ .2- .3	40.0	+ .6- .2	39.9	+ .6- .4	40.1	+ .3- .4	39.8	+ .3- .4	39.7	+ .7- .3	39.8	+ .4- .2
SERIES II																
80.....	Trace	-----	Trace	-----	Trace	-----	Trace	-----	Trace	-----	Trace	-----	Trace	-----	Trace	-----
100.....	Trace	-----	Trace	-----	Trace	-----	Trace	-----	Trace	-----	Trace	-----	Trace	-----	Trace	-----
140.....	7.9	+0.1-0.3	7.9	+0.2-0.1	7.4	±0.2	7.5	±0.2	7.3	+0.1-0.2	7.2	0	7.2	+0.1-0	7.3	+0.3-0.2
200.....	23.3	+ .4- .5	23.1	± .2	22.7	+ .2-0.4	23.0	+ .4-0.7	22.6	± .1	22.7	+0.3-0.6	22.8	+ .1-0	22.4	+ .4- .2
325.....	31.9	+1.0- .6	31.7	± .5	31.6	± .2	30.7	± .5	31.2	+ .4- .5	29.8	+ .2- .5	30.8	± .1	30.4	+ .2- .3
Pan.....	36.6	+1.0-1.3	37.2	+ .5- .4	37.7	+ .7- .5	38.3	+ .4- .6	38.4	+ .2- .1	40.0	+ .4- .5	38.8	+ .2-0.3	39.5	+ .6- .8

5, 10, 15, 20, 30, 40, and 60 minutes after the following treatments of the powder:

Series III—3 samples of each weight for each time period were oven dried for 1 hour at 110° C. prior to sieving.

Series IV—3 samples of each weight for each time period were exposed for 64 to 72 hours to a very humid atmosphere in a closed vessel (a desiccator) over water in which wicks were dipped to increase the evaporating surface.

Series V—The humidified samples of Series IV after sieving were mixed, dried for 5 hours at 110° C. and resieved.

The results are illustrated by the curves of figure 21, representing the pan fractions of the 100 gram samples. Similar results were obtained with the 25 and 50 gram samples. The value for the humidified samples, Series IV, are consistently lower than those of the dried material, Series III, by amounts corresponding to 3.5 to 6.5 percent of the original weight of the sample. Part of

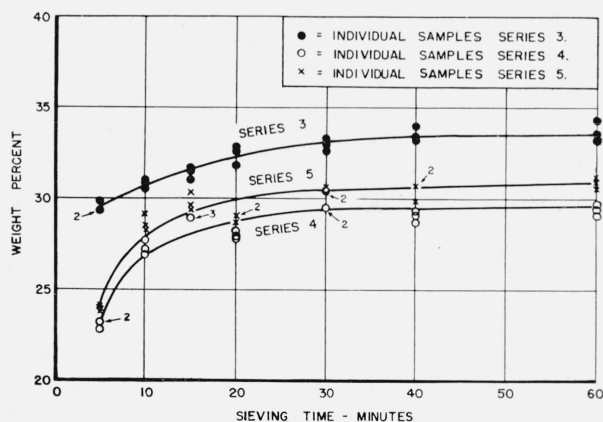


FIGURE 21.—Three series of sieve tests of sponge iron made with 100-gram samples all from the same "sample supply" after the following treatments of the powder: Series III, dried; Series IV, humidified; Series V, samples of Series IV dried and resieved.

this difference (approximately one-fourth) was recovered when the humidified samples were dried and resieved as shown by the position of the values for Series V. It will be noted that the values for individual samples of Series III are all quite close to the curve. The values for the

Series IV are more scattered, and the irregularities are matched by similar deviations in Series V.

The relative humidity in the humidification chamber undoubtedly was very high, probably saturated at times due to overnight or week-end changes in room temperature. Exposure to this atmosphere for 64 to 72 hours produced no change in the appearance of the powder except for a slight tendency to cake and to retain the shape of the dish in which the samples were contained. The gain in weight of the 100 gram samples, due largely to the presence of absorbed or condensed moisture, was 0.4 to 1.3 percent, average 0.8 percent. A large part of this weight increase was lost when the humidified samples were dried prior to resieving. The oxide content of the humidified samples after drying and resieving, as measured by the percentage loss in weight in hydrogen according to Metal Powder Association Tentative Standard 2-45T, was 1.2 percent. The hydrogen loss of samples taken from the original "sample supply" was 0.9 percent, indicating that the reducible oxide content of the powder was increased by 0.3 percent of the original weight of the sample during humidifying, sieving, drying and resieving.

All samples of the three series for each sieving period were taken from the same "sample supply" at the same time. The sieving for the three series was done concurrently, that is, all samples for each time period were sieved within the 72 to 96 hours required to humidify, sieve, dry, and resieve the samples of Series IV and V. These precautions eliminated the consideration of such variables, as the sampling error resulting from riffle splitting clogging of sieves or changes in sieve motion as major factors in causing the differences observed between the three series of tests. It must be concluded, therefore, that these differences resulted only from the exposure of the powder to a humid atmosphere.

Two additional series of sieve tests of sponge iron were made about 1 month after Series III, IV, and V were completed. The same sieves were used, but the samples, 25, 50, and 100 grams in weight, were taken from two different "sample supplies" riffle split within the same week. The samples were dried for 1 hour at 110° C and sieved for periods of 5, 10, 15, 20, 30, 40, and 60 minutes.

The pan fractions of the 100 gram samples are shown in figure 22 in comparison with those of

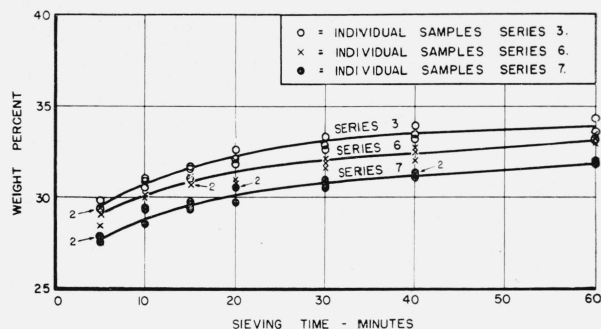


FIGURE 22.—Three series of sieve tests of 100-gram samples taken from three different "sample supplies" riffle cut from the same lot of sponge iron powder in the order Series III, Series VI, Series VII.

Series III. The difference between each succeeding series, in the order in which they were riffle-cut, amounts to about 1 percent of the original weight of the sample. The sampling, handling, and sieving for all of these tests were done in a heated laboratory during the winter months when the humidity was low and relatively constant. Changes in sieving characteristics resulting from exposure to such atmospheres should be small. It is believed, therefore, that the differences between Series III, VI, and VII resulted chiefly from sampling errors produced by repeated riffle cutting.

Variations similar to those obtained with sponge iron have been observed with electrolytic iron, electrolytic copper, and nickel in sieve tests made over extended periods of time. When a series of sieve tests of any of these powders was made on the same day or over a short period of time, the results for individual samples usually agreed within about 1 percent of the original weight of the sample. A similar series of tests made at another time using the same sieves and the same source of powder, again usually showed good agreement between the individual samples, but the average results often differed from those of the previous series by as much as 4 to 8 percent.

The variations observed in the sieve tests of these powders, extending over periods of 1 week to 8 months, are given in table 4. The maximum differences between corresponding fractions of electrolytic iron were as high as 5.6 percent with Sieve Set I (column 2) and 4.4 percent with Sieve Set 2 (column 4). With electrolytic copper, the differences were 7.8 percent (column 6) and 7.1 percent (column 8), respectively.



TABLE 4.—*Sieve tests of electrolytic iron, electrolytic copper, and nickel powders showing variation of results*  
[100-gram samples, sieved 30 min.]

Sieve No.	Weight percent retained											
	Electrolytic iron				Electrolytic copper				Nickel			
	Sieve Set I (25 tests)		Sieve Set II (20 tests)		Sieve Set I (33 tests)		Sieve Set II (48 tests)		Sieve Set I (7 tests) <sup>a</sup>		Sieve Set II (16 tests)	
	Mean	Deviation from mean	Mean	Deviation from mean	Mean	Deviation from mean	Mean	Deviation from mean	Mean	Deviation from mean	Mean	Deviation from mean
	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)
80	Trace	-----	Trace	-----	Trace	-----	Trace	-----	-----	-----	-----	-----
100	1.2	±0.1	1.3	+0.2-0.3	Trace	-----	Trace	-----	-----	-----	-----	-----
140	12.7	+ .6-0.7	10.5	+ .6- .7	6.2	+0.8-0.7	4.3	+0.9-0.8	2.3	+0 -0.1	1.4	+0.6-0.3
200	21.3	+1.3-1.0	24.4	+ .8- .6	16.4	+ .9- .8	19.1	+1.8-2.6	11.4	±0.1	12.5	+ .4- .7
325	20.3	+1.5- .7	23.9	+ .8- .6	20.6	+3.8-1.8	22.9	+3.0-2.4	12.5	± .3	15.4	+3.5-2.4
pan	44.0	+2.1-3.5	39.4	+2.4-2.0	56.1	+2.8-5.0	52.8	+4.8-2.3	73.4	± .2	70.3	+2.8-3.7

<sup>a</sup> These seven tests were made over a period of 1 week. All other tests covered periods of 6 to 8 months.

The data for nickel illustrate the agreement that may be expected in sieving tests made over a short space of time as well as the lack of agreement among tests made at different times. The results of seven tests with sieve Set I made over a period of about 1 week agree within 1 percent (column 10). The three series of tests with sieve Set II, using 6, 4, and 6 samples respectively, were made at different times over a period of about 8 months. The values within each series of tests made over a period of a few days, agreed within 1 percent, whereas differences between the series were as much as 6.5 percent.

Much of the data given in table 4 were obtained during the spring and summer seasons of the year. It is possible, therefore, that variations in the

atmospheric conditions to which the powders were exposed may have contributed to the variations noted in the sieve test results. A sampling error built up by several repetitions of riffle cutting might also account for part of the differences observed. Further work will be required under better controlled conditions, particularly with respect to sampling and humidity, before the relative effects of the two variables can be determined with certainty.

The average values of a large number of tests of electrolytic iron, electrolytic copper, and nickel, made with two different sets of certified sieves over long periods of time, are given in table 4. The differences between these averages (calculated to the third decimal place) are compared in table 5.

TABLE 5. *Sieve tests of electrolytic iron, electrolytic copper, and nickel powders showing differences in results obtained with two different sets of certified sieves<sup>a</sup>*

[100-gram samples, sieved 30 minutes]

Sieve No.	Weight percent retained															
	Sponge iron				Electrolytic iron				Electrolytic copper				Nickel			
	Mean, Sieve Set I (3 tests)	Mean, Sieve Set II (3 tests)	Difference of 2 means	Estimated standard error of difference	Mean, Sieve Set I (25 tests)	Mean, Sieve Set II (20 tests)	Difference of means	Estimated standard error of difference	Mean, Sieve Set I (33 tests)	Mean, Sieve Set II (48 tests)	Difference of 2 means	Estimated standard error of difference	Mean, Sieve Set I (7 tests)	Mean, Sieve Set II (16 tests)	Difference of 2 means	Estimated standard error of difference
	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)	(16)
80	Trace	Trace	-----	-----	Trace	Trace	-----	-----	Trace	Trace	-----	-----	Trace	Trace	-----	-----
100	Trace	Trace	-----	-----	1.172	1.260	0.088	.0.05	Trace	Trace	-----	-----	Trace	Trace	-----	-----
140	7.267	4.400	2.867	0.35	12.700	10.495	2.205	.12	6.152	4.294	1.858	0.09	2.286	1.431	0.855	0.10
200	22.600	24.400	1.800	.35	21.280	24.380	3.100	.22	16.352	19.065	2.713	.23	11.429	12.484	1.055	.10
325	31.167	33.000	1.833	.27	20.260	23.860	3.600	.17	20.630	22.927	2.297	.29	12.514	15.356	2.842	.77
Pan	38.400	37.867	.533	.11	43.956	39.425	4.531	.30	56.103	52.821	3.282	.43	73.371	70.314	3.057	.88

<sup>a</sup> The tests on nickel with Sieve Set I were made over a period of 1 week, tests on sponge iron over a period of 4 weeks, all others over periods of 6 to 8 months.

Included also are the averages of a few tests on sponge iron made with both sets of sieves within an interval of 4 weeks on samples from the same "sample supply". The maximum difference of the means for the two sets of sieves ranges from 2.9 to 4.5 percent and, although the same fractions are consistently larger or smaller for a given sieve mesh, the magnitude of difference varies with the different materials.

The standard error or standard deviation of the difference of the means,  $\sigma_D$ , given in columns 4, 8, 12, and 16 was estimated according to the formula

$$\sigma_D = (1/n + 1/m)^{1/2} (\Sigma X^2 + \Sigma Y^2/n + m - 2)^{1/2},$$

where

$n$  = the number of tests with sieve Set I

$m$  = the number of tests with sieve Set II

$$\Sigma X^2 = \Sigma X^2 - n\bar{X}^2$$

$$\Sigma Y^2 = \Sigma Y^2 - m\bar{Y}^2$$

$\Sigma X^2$  = the sum of the squares of the observations for sieve Set I at a given mesh

$\Sigma Y^2$  = the sum of the squares of the observations for sieve Set II at a given mesh

$\bar{X}$  = the arithmetic mean of the observations for sieve Set I at a given mesh

$\bar{Y}$  = the arithmetic mean of the observations for sieve Set II at a given mesh

The small magnitude of the estimated standard error in comparison to the difference of the means indicates that the difference between sieve sets was significant in spite of the wide scatter in individual values for tests made at different times.

Two new 325 mesh sieves were obtained from the manufacturer of Sieve Set I for use in either of the original sets if replacements should be necessary. These sieves were tested and certified by the National Bureau of Standards to conform to all specification requirements except material. The screen wire cloth was of monel metal instead of bronze, the latter being unavailable at the time of purchase. The differences in sieving results obtained when the new sieves were substituted in each of the original sets are shown in table 6. The tests with the new sieves (columns 3 and 6) were made on the same day. Those with the original Sieve Set I and Sieve Set II (column 1) were made 2 weeks before and 2 weeks later,

TABLE 6.—Sieve tests of sponge iron showing variations due to use of different sieves in same set

[The tests were made over a period of about 4 weeks in July. Average of 3 tests on 100-gram samples sieved 30 minutes]

Sieve No.	Weight percent retained								
	With original 325 mesh sieve		With new 325 mesh sieve (NBS No. 8887)		Difference from original (3) - (1)	With new 325 mesh sieve (NBS No. 8888)		Difference from original (6) - (1)	Difference between new sieves (6) - (3)
	Mean	Deviation from mean	Mean	Deviation from mean		Mean	Deviation from mean		
	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
SIEVE SET I									
80	Trace	-----	Trace	-----	-----	Trace	-----	-----	-----
100	Trace	-----	Trace	-----	-----	Trace	-----	-----	-----
140	7.3	+0.1-0.2	7.0	+0.1-0.2	-0.3	6.9	+0.1-0	-0.4	-----
200	22.6	±.1	22.4	+ .2- .4	-.2	22.6	±.1	0	-----
325	31.2	+ .4- .5	38.2	±.1	+7.0	41.0	+ .4- .2	+9.8	+2.8
Pan	38.4	+ .2- .1	32.0	+ .2- .1	-6.4	28.9	±.3	-9.5	-3.1
SIEVE SET II									
80	Trace	-----	Trace	-----	-----	Trace	-----	-----	-----
100	Trace	-----	Trace	-----	-----	Trace	-----	-----	-----
140	4.4	±0.6	5.2	+0.1-0	+0.8	5.1	0	+0.7	-----
200	24.4	±.6	23.5	+ .1-0.3	-.9	23.3	±0.1	-1.1	-----
325	33.0	±.2	38.6	+ .3- .2	+5.6	40.8	+ .1-0	+7.8	+2.2
Pan	37.9	±.2	32.2	±.1	-4.7	30.2	±.1	-7.7	-2.2

respectively. All samples were taken from the same "sample supply". It will be noted that the fractions larger than 200 mesh agreed within 1.2 percent with any given sieve assembly. The new 325 mesh sieves retained 7.0 and 9.8 percent more material, respectively, than the sieve they replaced in Sieve Set I. With Sieve Set II, the differences were 5.6 percent and 7.8 percent with corresponding changes in the pan fraction. The amounts retained by the two new sieves differed by 2.8 percent when used with Sieve Set I and 2.2 percent with Sieve Set II.

In connection with these differences it is interesting to compare the average openings of the several sieves. These sieve openings, as measured during the certification tests, are given in table 7.

The variation in the average opening permitted in 325 mesh sieves by ASTM Specification E-11 is  $44 \text{ microns} \pm 7 \text{ percent}$ , or from 41 to 47  $\mu$  inclusive. The average openings of the four 325 mesh sieves range, in the order given in table 7, from a value near the upper limit to one at the

lower limit. The weight of the fractions retained by these sieves vary in the reverse order, as would be expected, the sieve with the largest average opening retaining the smallest quantity, the sieve with the smallest average opening retaining the largest quantity. The effect of seemingly negligible differences in sieve aperture on sieve tests is illustrated by the results obtained with sieves 8887 and 8888. Although the sieves differ by only 1  $\mu$  between the shoot wires, the fractions retained by Sieve 8888, with the smaller aperture, were 2.2 to 2.8 percent greater than those retained by the other sieve.

Variations of such magnitude in sieving results with different sieves are not peculiar to metal powders; similar variations observed by many workers with nonmetallic powders were mentioned in the introduction to this paper.

## VI. Size Distribution within Fractions

The particle size distribution of the fractions retained on the several sieves was determined by microscopic measurement. Xylene containing a few drops of cedar wood oil was used as a dispersing agent in making the slides. Measurements were made on the image of the particles projected upon the ground glass of a camera at a magnification of  $\times 50$ . The projected dimension measured was the one which, in the opinion of the observer, would control the passage of the particle through a sieve opening.

The controlling dimension was easily determined when, as was usually the case, the projected image was circular, roughly rectangular or elliptical. However, when very irregular particles, such as the "tree-shaped" electrolytic copper particles shown in figure 5, were measured, selection of the controlling dimension was difficult. In such cases the branches were considered a part of the dimension when they were small or close together, as at *A* and *B* (fig. 5). When the branches were widely separated, as at *C*, the dimension was measured from the fork of the branch. The controlling dimensions obtained for the three particles by this procedure (between 5 and 6 mm at  $\times 50$  magnification) are in line with the dimensions of the other particles of the sample.

The particle size distribution of the five sieve fractions of sponge iron larger than 325 mesh is shown in figure 23. The fractions retained by the

TABLE 7.—Average opening of sieves as measured in certification tests

Sieve No.	NBS Certi- fication No.	Average opening <sup>a</sup>	
		Between warp wires	Between shoot wires
ORIGINAL SIEVE SET I			
		$\mu$	$\mu$
80	8805	186	174
100	8806	143	148
140	8807	107	102
200	8808	78	75
325	8809	46	46
ORIGINAL SIEVE SET II			
80	8889	185	174
100	8890	144	140
140	8891	104	107
290	8892	77	73
325	8893	43	45
NEW NO. 325 SIEVES			
325	8887	41	43
325	8888	41	42

<sup>a</sup> These measurements were made under the supervision of L. V. Judson of the Metrology Division of the National Bureau of Standards who states that the measurements were made with an accuracy sufficient to determine whether or not the openings were within the limits permitted by specifications. It is not likely that the errors are in excess of about 1  $\mu$ .

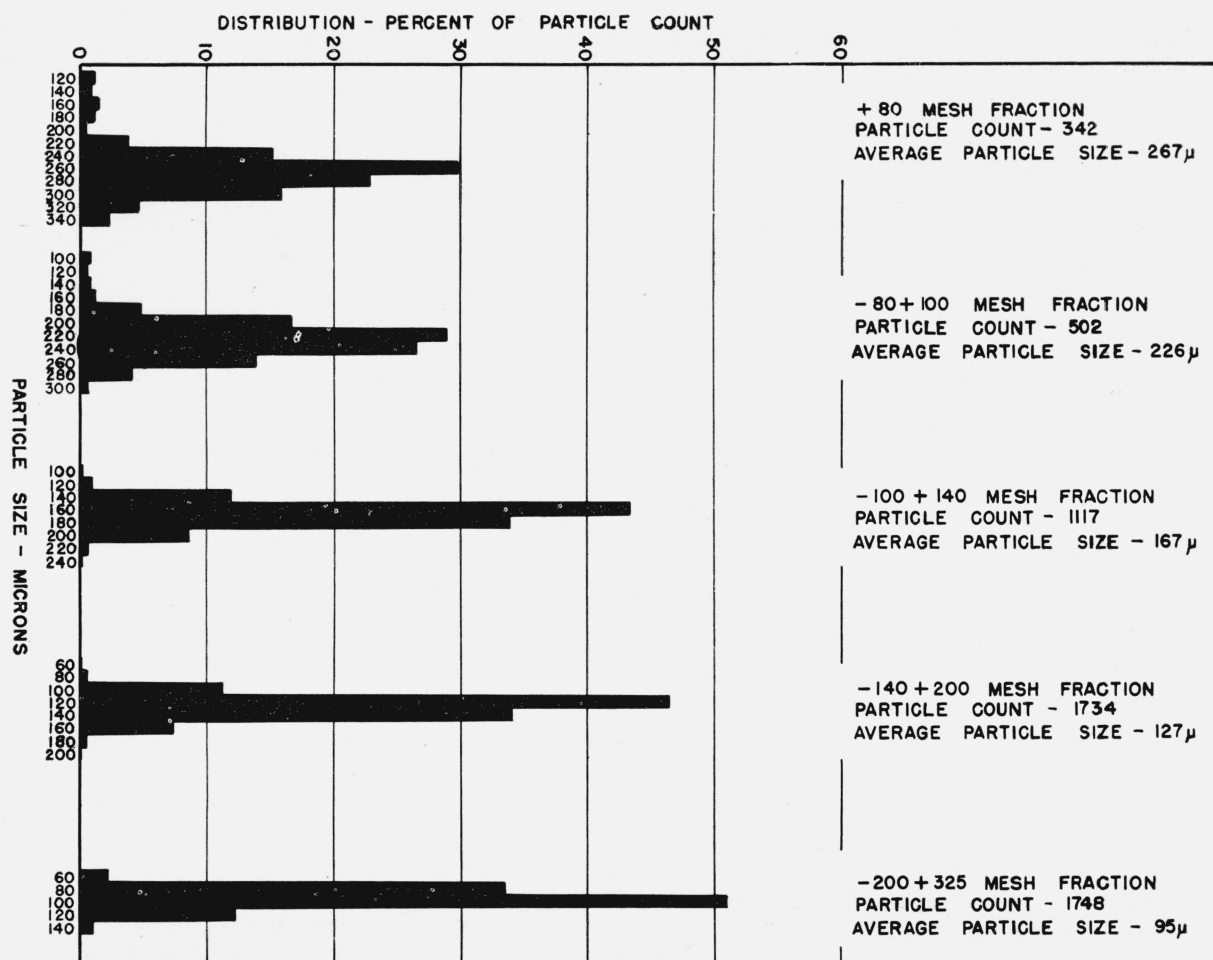


FIGURE 23.—Particle size distribution of sieve fractions of sponge iron measured microscopically at a magnification of  $\times 50$  (accuracy  $\pm 10$  microns).

80 and 100 mesh sieves contain particles ranging in size from class mark 100 to class mark  $340\mu$ . The range in size distribution decreases considerably with the finer sieves. However, even in the  $-200+325$  mesh fraction the range in particle size is over  $80\mu$  (from class mark 60 to class mark  $140\mu$ ) with over 50 percent of the particles between 90 and  $110\mu$ .

The average particle size of a sieve fraction is often described in the literature as the mean of the nominal openings of the sieves which retained, and which just passed the fraction. The average particle size of sieved fractions of the metal powders listed in table 1, and the mean apertures of the passing-retaining sieves are given in table 8. Measurements were made at magnifications of  $\times 50$  and  $\times 100$ , with accuracies of  $\pm 10$  and  $\pm 5\mu$ , respectively. The average particle size of the

TABLE 8.—Average particle size of sieve fractions of metal powders as measured microscopically at magnifications of  $\times 50$  and  $\times 100$  with accuracies of  $\pm 10$  and  $\pm 5$  microns respectively

Sieve fraction	Mean aperture of passing-retaining sieves	Average particle size, controlling dimension						
		Sponge iron	Electrolytic iron	Electrolytic copper	Nickel	Zinc <sup>a</sup>	Tin	Lead
+80	$\mu$	$\mu$	$\mu$	$\mu$	$\mu$	$\mu$	$\mu$	$\mu$
-80+100	163	267	278	271	246	219		228
-100+140	127	226	238	186	228	157	194	190
-140+200	89	167	173	136	178	129	142	126
-200+325	59	127	134	112	124	92	90	98
		95	96	86	86	63	64	68

<sup>a</sup> Measured at  $\times 100$ . All other measurements made at  $\times 50$  magnification.

fractions vary greatly with the different materials. The values for the atomized powders, zinc, tin, and lead, composed of roughly spherical particles with



the three dimensions approximately equal, are close to the mean of the sieve apertures. The plate-like or very irregularly shaped particles of electrolytic iron, sponge iron, copper, and nickel have controlling dimensions considerably greater than the mean of the apertures. This tendency for the projected dimensions of particles, particularly irregularly shaped particles, to exceed the average aperture of the passing-retaining sieves has been noted by other investigators [17, 18, 19, 20].

## VII. Discussion of Results

The results of the sieving tests indicate that the sieving rates of metal powders vary considerably with different sieves and with different materials, that appreciable variations in results may be obtained when the same powder is sieved at different times with the same sieves and that sharp or complete separation into fractions cannot be attained by sieving, at least in a reasonable length of time. Hence, selection of the proper sieving time for a given material would appear difficult. However, the results also show that reproducible values can be obtained when certain sampling errors and other variables affecting the physical condition of the powder, such as exposure to high humidities, can be eliminated or controlled. Under these conditions the scatter in values for samples of the same weight sieved for the same time apparently is independent of the time of sieving (see figs. 17 to 22). Any time between 5 minutes and 2 hours could be selected without seriously affecting the reproducibility, provided the other conditions are fixed. As prolonged sieving would increase the proportion of particles passed by oversized openings, it would seem advisable to select a sieving time near the end of the period of rapid sieving rate, that is, after the separation of the definitely undersized particles. The time required for sponge iron would be 15 to 30 minutes, depending upon sample size and other conditions.

Two variables seemed to cause the most serious differences observed in the results of sieve tests made at different times with the same sieves and the same powder. One of these was a sampling error produced in riffle cutting, the other the result of exposure of the powder to high humidity. The sample error in riffle cutting can be attributed largely to the loss of fines as dust during the

operation. Evidently this is not a good method to use for sampling limited supplies of metal powders, particularly if the cutting operation is to be repeated, for then the sampling error becomes cumulative. A noncumulative method, possibly the use of a sample thief, might improve the reproducibility.

The effect of exposure to humid atmospheres on the sieving characteristics of some metal powders as demonstrated by the tests on sponge iron and suggested by the results on electrolytic iron, electrolytic copper, and nickel, poses difficult problems of storage and handling. With wrought metals it is known that at ordinary temperatures and low humidity corrosion is very slow or non-existent. Polished samples of sheet steel, for instance, have been stored for many years in an atmosphere of about 50 percent relative humidity (over a saturated solution of calcium nitrate) without evidence of rust. However, with finely divided metal powders oxidation or corrosion may proceed at much lower humidities. Relatively small temperature changes also may have an effect. For example, it has been noted that some very fine powders, notably electrolytic copper and tin (89 to 91 percent through 325 mesh) change color when heated at 110° C for drying.

The problem posed by the differences in results obtained with different certified sieves also is difficult. There seems to be little hope that sieves can be manufactured, by present methods at least, to tolerances sufficiently close to eliminate the differences caused by minute variations in the dimensions of the average openings. The long continued and successful use of sieve analyses in the production and utilization of powdered substances shows that present manufacturing tolerances and calibration methods are adequate for control purposes at least. For research work, a more accurate method of evaluating the differences obtained with sieves that vary within specification tolerances would be desirable.

It should be pointed out that the variations in results obtained with different sieves are not as great as is indicated by comparison of values on the basis of the nominal opening. For example, in figure 24, cumulative curves of the data given in table 6 are shown in which the percentage by weight passing the several sieves is plotted against the size of the average openings as determined during the certification tests. It will be noted

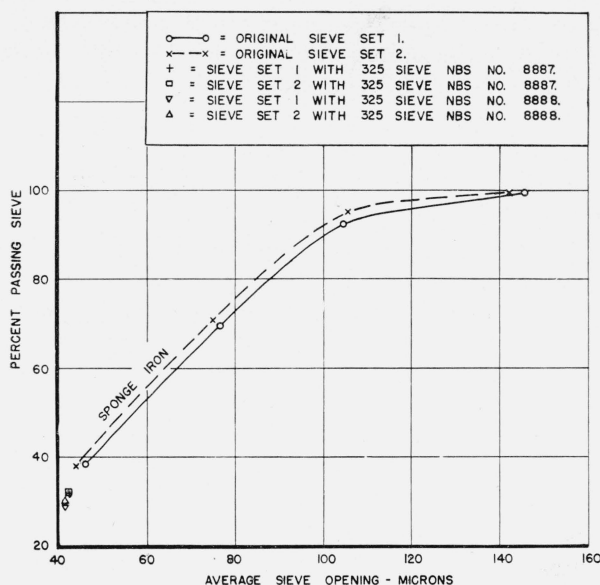


FIGURE 24.—Cumulative weight distribution curves for sponge iron using the actual size of the average opening of the different certified sieves instead of the nominal opening as the bases of comparison.

100-gram samples sieved for 30 minutes.

that the differences between sieves are considerably less than they would be if the comparison were made on the basis of the nominal openings.

Possibly still closer agreement would be found if the variations in size of sieve openings were taken into consideration after the method of Weber and Moran [21]. This method consists in measuring random openings microscopically. Two sieves are said to be identical if the average openings and the standard deviations are the same. The percentage standard deviation or coefficient of variance  $V=100 \sigma/X$ , where  $\sigma$  is the standard deviation and  $X$  the average opening, is used as the measure of sieve equivalence. An empirical formula based on the sieving time and the coefficient of variation has been applied to evaluate the effective openings of different sieves used in sieve analyses of soda ash and sodium bicarbonate.

In the cement industry, the No. 200 sieves used in accordance with ASTM Standard C184-44 for fineness tests of hydraulic cement [10] are calibrated against a standard sample of cement prepared by the National Bureau of Standards.

Hatch [17], using crushed limestone, measured the dimensions of sieved particles microscopically and, by statistical methods, calculated the size distribution by weight. The calibrated size of a

sieve was taken as the median or geometric average size by weight of the material retained on the sieve.

Fagerholt [22], working with ground sand and feldspar, found that the particle size to which a result obtained by sieving in the time,  $t$ , should be referred in determining the cumulative weight distribution is the average particle size of the fraction passing the sieve by continued sieving under the same conditions to the time  $3t$ . This particle size is determined by counting the number of particles in a weighed portion of the fraction obtained during the interval  $t$  to  $3t$ .

It is possible that these or similar methods of calibration could be applied to sieve analyses of metal powders. Because of the wide variations in particle shape and particle size distribution resulting from many different production methods, it is not to be expected that any one formula or standard material or calibration factor can be devised that will be equally applicable to all metal powders. However, even limited application to a powder from a given source or possibly to restricted classes of powders, such as iron made from reduced mill scale or electrolytic copper or atomized zinc, would be of value. These applications and limitations must be determined by further investigation that should include further study of the effect of humidity.

## VIII. Summary

In sieve tests of sponge iron, electrolytic iron, electrolytic copper, and nickel, reproducible results were obtained only when certain variables were controlled. One factor was a cumulative sampling error resulting from repeated riffle cutting of limited powder supplies. Another variable affecting the sieving characteristics of the powders was exposure to different atmospheric conditions.

The effects of these variables was demonstrated by tests on sponge iron. Differences obtained on successive riffle cut samples were of the order of 1 percent of the original weight of the sample. The effects of humidity were considerably greater, differences of 3 to 6 percent being observed between sieve tests of dried samples and samples exposed to high humidities.

Variations of considerable magnitude were observed in the results of sieve tests of the same metal powder when sieved under comparable

conditions with different sets of certified sieves. With sponge iron, these differences were as high as 9.8 percent when comparisons were made on the basis of the nominal sieve opening. When compared on the basis of the actual size of the average opening as measured during the certification tests, the differences were reduced considerably.

Further investigation of possible methods of eliminating, controlling, or evaluating the effects of all of these variables is desirable.

Grateful acknowledgment is made to Louis V. Judson for his constructive criticism of testing methods and interpretation of results; to C. M. Saeger who was formerly connected with the investigation; to W. Earl Lindlie who assisted in the preparation of graphs; and to Eleanor M. Crook, Ruth K. Kimble, and Mary E. Steinbaugh who assisted in the sieve testing and microscopic work.

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